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SUPERCONDUCTING MATERIAL AND METHOD OF SYNTHESISField of the Invention

5 The present invention relates to a superconducting compound and to a method of synthesising a superconducting compound, and, particularly, but not exclusively, a superconducting compound based on magnesium diboride and a method of synthesis thereof.

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Background of the Invention

Significant progress has been made in improving the critical current density (J_c) in various forms of MgB_2 since superconductivity in this compound was discovered [1]. MgB_2 exhibits the superconducting characteristics and physics of BCS-type LTS (low-temperature superconductor) materials, as evidenced for example by a significant isotope effect [2]; however, its critical temperature (T_c) is more than twice those of the presently used superconductors Nb_3Sn and Nb_3Al , and more than four times that of the present LTS workhorse, $NbTi$. The importance of MgB_2 lies in its simple crystal structure, high critical temperature T_c , high critical current density (J_c), and large coherence length (hence transparency of grain boundaries to current flow). These properties of MgB_2 offer the promise of important large-scale and electronic device applications. High J_c at a level of 10^5 A/cm^2 to 10^6 A/cm^2 at 20 K to 30 K for MgB_2 wires have been reported by several groups [3-8]. However, J_c drops rapidly with increasing magnetic field. In all the studies on wires and bulk made from MgB_2 , J_c decreased more than 90% of its zero field value at 3 T within this temperature range due to the poor pinning ability of this material. If MgB_2 is to be useful in high fields, the flux pinning strength must be improved. Attempts to enhance flux pinning have resulted in an encouraging improvement in irreversibility fields

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(H_{irr}) and $J_c(H)$ performance in high fields by oxygen alloying in MgB₂ thin films [9,10] and by proton irradiation of MgB₂ powder [11].

However, there has been some conjecture as to whether 5 effective pinning centres can be introduced into MgB₂ bulk or wires by a normal process such as chemical doping.

Several attempts have been made to improve flux pinning using chemical doping, but the results remain controversial. Furthermore, the results for doping into 10 MgB₂ are largely limited to addition rather than substitution. Zhao et al, have doped MgB₂ with Ti and Zr, showing improvement of J_c in self field [12]. However, there is evidence for improved pinning as the J_c drops off rapidly with increasing field ($H_{irr} = 4T$ at 20K). Recently, 15 Wang et al doped MgB₂ using nanoparticles [13]. The results showed an improvement of irreversibility field (H_{irr}) at 4.2K for the doped sample. However, H_{irr} for the doped samples is not as good as the undoped ones at 20K. Cimerle et al., found that doping with a small amount of Li, Al 20 and Si showed some increase in J_c , but there is no improvement in H_{irr} [14].

Summary of the Invention

25 In accordance with a first aspect, the present invention provides a superconducting material of formula MgB_xSi_yC_z where X is a number in the range between 0 to 2, Y is a number in the range between 0 to 1 and Z is a number in the range 0 to 1, and where the sum of X, Y and 30 Z is greater than or equal to 2.

Preferably, X = 1 to 2, Y = 0.05 to 0.5 and Z = 0.05 to 0.5.

More preferably, X = 1.2 to 1.8, Y = 0.1 to 0.3 and Z = 0.1 to 0.3.

35 Advantageously, the factors of critical current density, irreversibility field and flux pinning properties of MgB₂ are significantly improved by chemical doping with

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SiC, potentially paving the way for MgB₂ to replace the current market leaders NbTi and Ag/Bi2223.

It will be understood that a superconducting material in accordance with the present invention may preferably be 5 made into superconducting bulk, wires, thin films and various articles and devices for any superconducting application.

In accordance with a second aspect, the present invention provides a superconductor incorporating the 10 superconducting material of the first aspect of the invention.

In accordance with a third aspect the present invention provides a method of synthesising the superconducting material of the first aspect of the 15 present invention, comprising the step of utilising starting materials Mg, B, Si and C. Preferably, these are in powder form. Preferably, the powders consist of nanoparticles.

In accordance with a fourth aspect, the present 20 invention provides a method of synthesising the superconducting material of the first aspect of the invention comprising the steps of utilising starting materials Mg, B and SiC. Preferably, these are in powder form. Preferably, the powders consist of nanoparticles.

In accordance with a fifth aspect, the present 25 invention provides a method of synthesising a superconducting material in accordance with the first aspect of the invention comprising the step of utilising starting materials MgB₂ and SiC or Si and C. Preferably, 30 these are in powder form. Preferably the powders consist of nanoparticles.

More broadly, in accordance with a sixth aspect, the present invention provides a method of producing a superconducting material, comprising the step of adding 35 silicon carbide to a superconducting material.

Preferably, the silicon carbide is added by way of addition.

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Preferably, the silicon carbide is added by way of substitution.

The silicon carbide may be added both by substitution and addition to the superconducting material.

5 In accordance with a seventh aspect, the present invention comprises a superconducting material including a silicon carbide codopant.

In an eighth aspect, the present invention provides a method of manufacturing a material capable of functioning 10 as a superconductor, comprising the steps of,

- mixing elemental magnesium and elemental boron with an amount of one or more of the group comprising silicon carbide and titanium carbide, and
- 15 heating the powders to sinter the powders into a material capable of functioning as a superconductor.

In a ninth aspect, the present invention provides a method of manufacturing a material capable of operating as 20 a superconductor, comprising the steps of,

- mixing elemental magnesium and elemental boron with an amount of one or more of the group comprising elemental silicon, elemental titanium and elemental carbon, and
- 25 heating the mixture to sinter the mixture into a material capable of functioning as a superconductor.

Preferably, the mixture is heated to a temperature in the range from 650° to 2000°C. More preferably, the 30 temperature is in the range 750°-900°C.

Preferably, the elements are provided in a powder form.

Preferably, the powders consist of nanoparticles.

Preferably, the powders are groove-rolled into a tube 35 manufactured from a material of one or more of the group comprising iron (Fe), copper (Cu), nickel (Ni) and stainless steel tube prior to heating the mixture.

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Preferably, the method comprises the further step of cooling the resultant material to the temperature of liquid nitrogen, to render the material capable of superconducting.

5 In a tenth aspect, the present invention provides a superconducting material of the formula of formula $MgB_xTi_yC_z$ where X is a number in the range between 0 to 2, Y is a number in the range between 0 to 1 and Z is a number in the range 0 to 1, where the sum of X, Y and Z is greater than or equal to 2, and X is greater than 0.

Brief Description of the Drawings

Preferred features and advantages of the present 15 invention will become apparent from the following description of examples thereof, by way of example only, with reference to the accompanying figures, in which;

Figure 1 is a graph depicting the Critical transition temperature (T_c) measured using magnetic susceptibility versus temperature for $MgB_2(SiC)_x$ where $x = 0, 0.055, 0.11, 0.22$ and 0.33 .

Figure 2 is an image of X-ray diffraction patterns for the undoped and SiC-doped samples of a superconducting material in accordance with an embodiment of the present 25 invention.

Figure 3 is a graph depicting the lattice parameters 'a' and 'c' plotted as a function of the SiC content x of a superconducting material in accordance with an embodiment of the present invention.

Figure 4 is a graph depicting the effect of SiC doping on the critical current density as a function of applied magnetic field (i.e. the $J_c - H$ dependence) at a temperature of 5 K (graph a), 10 K (graph b) 20 K (graph c) and 30 K (graph d) for five samples at doping level 35 ranging from $x=0$ to $x=0.33$.

Figure 5 is a graph which depicts the comparative values of critical current density($J_c(H)$) as a function of

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applied magnetic field for SiC doped samples of a superconducting material in accordance with an embodiment of the present invention at a doping value of $x=0.115$ at 20K with those for Ti doped [12], Y_2O_3 doped [13], thin 5 film with strong pinning [11] and Fe-sheathed tape [15], which represent the state-the-art performance of MgB_2 in various forms.

Figure 6 is a graph depicting the transport critical current density as a function of applied magnetic field 10 for an SiC doped sample of a superconductor in accordance with an embodiment of the present invention, for a value of $x=0.11$ measured at a temperature of $T=5\text{K}$, compared to prior art MgB_2 superconductors measured at a temperature of $T=4.2\text{K}$

15 Figure 7a and 7b are a transmission electron microscope (TEM) images depicting the high density dislocations within the grains and Figure 7c is an energy dispersive X-ray (EDX) analysis map showing the incorporation of C and Si into the grains of MgB_2 .

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Description of Examples and Preferred Embodiments

The superconducting composition and the processes for synthesis of the materials of the present invention can 25 significantly enhance J_c and flux pinning. The results which are described hereinbelow demonstrate that the claimed formula can be used for fabrication of superconductors for high-field applications, as chemical doping is a readily achievable and economically viable 30 route to introduce effective flux pinning.

MgB_2 pellet samples were prepared by a reaction in-situ method which has been previously described [16]. Powders of magnesium (99% purity) and amorphous boron (99% purity) were well mixed with a SiC additive with the 35 weight ratio of $(\text{Mg}+2\text{B})(\text{SiC})_x$ where $x = 0, 0.055, 0.11, 0.22$ and 0.33 for samples 1 to 5 respectively (Table 1). The mixed powders were loaded into Fe tubes. The composite

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tubes were groove-rolled, sealed in a Fe tube and then directly heated at preset temperatures to 950°C for 3 hours, in the presence of flowing high purity Ar. This was followed by quenching with liquid nitrogen. Table 1 gives 5 the samples parameters and selected results of T_c and H_{irr} .

The magnetization of samples was measured over a temperature range of 5 to 30 K using Magnetic Property Measurement System (MPMS) and a Physical Property Measurement System (PPMS, Quantum Design) in a time-varying magnetic field of sweep rate 50 Oe/s and amplitude 10 5T and 9T, respectively. A magnetic J_c can be derived from this measurement.

Figure 1 depicts the transition temperature (T_c) and transition width ΔT_c for the doped and undoped samples 15 determined by AC susceptibility measurements. The T_c onset for the undoped sample (38.6 K) is the same as reported by a number of groups. For the doped samples, the T_c decreases with increasing doping level. It is striking to note that despite the large amount of non-superconducting phases 20 present, the T_c only drops 2.6K at an SiC doping level of $x=0.33$ (33at% of B). In contrast, the T_c was depressed for almost 10K by 7% C substitution for B in MgB_2 [17] and 0.5at% Si substitution reduced T_c for about 0.5K [14]. These 25 results suggest that the higher tolerance of T_c of MgB_2 to SiC doping is attributable to the co-doping of C and Si because the average size of C (0.077nm) and Si (0.117nm) is similar to that of B (0.097nm). It is evident that the co-doping with SiC counterbalanced the negative effect on T_c of the single element doping.

30 Figure 2 depicts x-ray diffraction patterns for SiC doped and undoped samples of an MgB_2 superconductor. The X-ray scans were recorded using $Cu_K\alpha=1.5418\text{\AA}$, and indexed within the space group P6/mmm. For the in-phase reflection which occurs in Figure 1 between $2\theta=33^\circ$ and $2\theta=34^\circ$ 35 (indexed as (100)), the centroid of the peak clearly shifts to higher 2θ values with increasing x , while simultaneously the centroid of the peak occurs between

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2 θ =51° and 2 θ =52°, (indexed as (002)), and the shift is marginal to higher 2 θ values with increasing x. The lattice parameters, a and c of the hexagonal AlB₂-type structure of MgB₂ were calculated using these peak shifts
5 as shown in Figure 3. Note that the MgB₄, MgO and MgB₄O₇ peaks increased significantly with increasing SiC. The continuous decrease of 'a' with increasing SiC doping level indicates that B was substituted by C and Si. C substitution for B reached saturation at 7at% of B [19]
10 while the co-doping of Si and C for B raised the saturation level. At x=0.055, the sample consists of a major phase with MgB₂ structure and minority phases: MgB₄, MgBO and MgO. The appearance of MgB₄ may be attributable to the substitution of SiC for B position, resulting in an
15 excess of B. Some extra B was incorporated into MgO to form MgBO. It is also noted that all three non-superconducting phases increased with increasing SiC dopant. At x=0.22, the amount of non-superconducting phases exceeded MgB₂. The extra oxygen may be brought in by
20 the SiC dopant which absorbed moisture or oxygen during storage. There are no SiC peaks indexed up to a value of x=0.33.

Note that in figure 3 the axis decreases with increasing SiC dopant and did not reach saturation at
25 x=0.33. The total variation of the axis from x=0 to x=0.33 is 0.012A. In comparison, with single element doping with C, the axis reached a plateau at a C content of 7at% of B where the variation of the axis is 0.016A. This indicates that co-doping of Si and C into MgB₂ substantially reduced
30 the variation of axis due to the counterbalance effect of Si and C. This also explains why the T_c drops very slow with increasing SiC dopant.

Figure 5 shows the J_c(H) curves for doped and undoped samples at temperatures of 5 K (graph a) and 10 K (graph
35 b). These results show the following striking features. The J_c(H) curves for undoped samples shows a crossover with those for all the doped sample at higher fields. Although

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SiC doping at x values of greater than 0.22 caused a reduction of J_c at low fields, it is important to note that the J_c for all doped samples drops with increasing field much more slowly than for the undoped sample. In particular, J_c curves for doped samples with x values of 0.22 and 0.33 show an exponential relationship with the applied magnetic field up to the measurement field limit, while the J_c curve for the undoped sample shows a rapid downward bend. The J_c for the doped sample at a value of $x=0.11$ reached $1.3 \times 10^5 \text{ A/cm}^2$ at 4T and increased to a value more than 30 times that of the undoped sample at a field strength of 6T and a temperature of 5K. It is also interesting to note that at temperatures of 5K and 10K, $J_c(H)$ for higher doping level samples (4 and 5) declined faster than those of low doping samples (2 and 3). However, at 20K all doped samples showed nearly parallel $J_c(H)$ curves while at 30K, the $x=0.33$ doped sample showed an even slower J_c drop with field relative to the other samples, indicating the presence of a stronger pinning enhancement effect at higher temperatures.

Figure 4 depicts a comparison of the J_c - H behaviour for an $x=0.11$ SiC doped sample with data reported from literature at various temperatures. It is evident that despite the low density and unoptimised composition, the J_c for the SiC doped sample drops slower than other element doped samples [12, 13], the best Fe/MgB₂ tape [15] and even close to the thin film with strong pinning [11]. At 20K, the sample with $x=0.11$ has a J_c value of $18,000 \text{ A/cm}^2$ at 4T, 100 times the control sample which was made at the same batch, 8 times that of the state-the-art Fe/MgB₂ tape [15]. These are the best J_c values ever reported for bulk and wires made under normal conditions. The H_{irr} for $x=0.11$ doped sample is 6T at 20K and 8T at 10K, compared to 4T and 6T of the undoped one, respectively. A further comparison is shown in Figure 6, where the results of transport current measurements are shown for one of the most optimal SiC-doping levels and the best MgB₂ samples

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found in the literature. As can be seen, there is a striking difference between the two curves. Moreover, the SiC-doped sample is measured at a temperature of 5K, whereas the other samples are measured at a temperature of 5 4.2K. In other words, the actual enhancement induced by SiC-doping is even larger than the best MgB₂ samples found in the literature.

Regarding the mechanism of the enhancement of J_c at higher fields, it is necessary to recognize the special 10 features of SiC doping.

Firstly, in contrast to previous work on doping for improving J_c [12-14], SiC doping has no densification effect, as evidenced by the fact that the density of doped samples is 1.2G/cm³, independent of doping level. This is 15 understandable because SiC has a very high melting point and would not act as a sintering aid at temperatures in the range of 800°C to 950°C.

Secondly, SiC doping takes place in the form of substitution and/or addition while in the prior art [14-20] 16] the element doping is in the form of additives, which are not incorporated into the lattice structure. Doping MgB₂ with Ti and Zr showed an improvement of J_c in self field and 4K [12]. However, there is evidence for improved pinning as the J_c drops off rapidly with increasing field 25 (H_{irr} = 4T at 20K). Doping MgB₂ using Y₂O₃ nanoparticles showed an improvement of irreversibility field (H_{irr}) at 4.2K, but H_{irr} for the doped samples is not as good as the undoped ones at 20K [13]. Cimerle et al., found that 30 doping with a small amount of Li, Al and Si showed some increase in J_c, but there is no improvement in H_{irr} [14]. It is evident that the additive pinning is more effective at low temperatures while the additives at the grain boundaries decouple the grains at high temperatures.

The applicant postulates that there are two potential 35 pinning mechanisms in the SiC doped samples. The first is intrinsic pinning due to substitution and impurity pinning (or a combination thereof). The high fraction of

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substitution by both Si and C can result in lattice defects, which are capable of acting as effective pinning sites which are intrinsic in nature and independent of temperature. The high content of MgO and other impurity 5 phases in the SiC doped samples could also be potential pinning centers, consistent with the results obtained from a thin film with strong pinning where the ratio of Mg:B:O reached 1.0:0.9:07 [11]. the applicant has attempted to dope fine particle MgO into MgB₂. However, the results did 10 not show any improvement in J_c. From this result, it is theorised that the manner in which the impurities are introduced may be critical. When SiC reacts with liquid Mg and amorphous B at the sintering temperatures, the 15 nanoparticles may act as nucleation sites to form MgB₂ and other phases. Some nanoparticles may be included within the grains as inclusions. Thus, the reaction induced products are highly dispersed in the bulk matrix. These arguments are supported by the study of microstructures. TEM examination revealed that the grain size is smaller 20 than 100nm. The EDX analysis results showed that the Mg:Si ratio is identical across the entire sample area, indicating that the phase distribution is homogeneous. The results of the present work suggest that a combination of substitution and highly dispersed additives induced 25 through the substitution is responsible for the enhanced flux pinning in SiC-doped MgB₂. The substitution induced defects and order parameter fluctuation may play a more important role than the impurity pinning.

It should be noted that the density of the samples is 30 only about 1.2 g/cm³. Thus, the J_c values for both the doped and undoped samples are far from optimum. The applicant anticipates that a higher J_c and better flux pinning enhancement can be achieved if the density of the samples is further improved.

35 From the study of effect of the purity of the precursor materials, it is noted that even 95% pure B degraded the J_c appreciably. Therefore, it is necessary to

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use high purity B (98% or above). The cost for B increases significantly with increasing purity. The main cost for making MgB₂ conductors will be the high purity B. Since C and Si are abundant, inexpensive and readily available materials, then if a portion of B can be replaced by co-doping with C and Si or SiC, the overall cost for making MgB₂ conductors will preferably be reduced. Furthermore, the SiC doping has already shown a significant benefit in enhancing flux pinning. It is evident that it is advantageous for MgB₂ conductors to be made using a formula of MgB_xSi_yC_z where x+y+z≥2, instead of pure MgB₂.

In summary, the applicant contends that the critical current density, irreversibility field and flux pinning properties of MgB₂ in bulk form can be significantly improved by a readily achievable and economically viable chemical doping with SiC, which may allow MgB₂ to potentially replace Nb-Ti, the current market leader in superconducting materials.

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Table 1

Sample No	SIC wt%	At. Ratio MgB ₂ :SiC	Density G/cm ³	T _{c(0)} (K)	H(T) 10K	H(T) 20K	H(T) 25K	H(T) 30K
1	0	1: 0	1.20	38.6	6.2	4.0	3.6	2.2
2	5	1: 0.055	1.21	37.8	7.2	5.1	4.0	2.5
3	10	1: 0.11	1.22	36.8	8.0	5.8	4.5	2.5
4	20	1: 0.22	1.17	36.6	5.5	3.7	3.5	1.8
5	30	1: 0.33	1.30	36	5.2	4.0	3.2	2.5

Example 1: Synthesis of (Mg+2B)_{1-x}(SiC)_x through nanoparticle SiC doping into MgB₂

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(Mg+2B)_{1-x}(SiC)_x samples were prepared by a reaction in-situ method. Powders of magnesium (99% purity) and amorphous boron (99% purity) were well mixed with SiC additive with the atomic ratio of (Mg+2B)_{1-x}(SiC)_x where x = 0, 0.057, 0.117, 0.23 and 0.34 for samples 1 to 5 respectively

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(Table 1). The mixed powders were loaded into Fe tubes. The composite tubes were groove-rolled, sealed in a Fe tube and then directly heated at preset temperatures to 950°C for 3 hours, in the presence of flowing high purity Ar. This was followed by quenching to liquid nitrogen temperature. Table 1 provides information, with samples 1, 2, and 3 being the undoped and doped with 5wt% and 10wt% MgO respectively.

The magnetization of 1.0 x 1.0 x 0.8 mm³ samples was measured over a temperature range of 5 to 30 K using a Physical Property Measurement System (PPMS, Quantum Design) in a time-varying magnetic field of sweep rate 50 Oe/s and amplitude 9T. A magnetic J_c can be derived from the height of the magnetization loop ΔM using a suitable variant of the "semi-Bean" relationship $\Delta M = k J_c d$, where k is a constant and d is the thickness that the sample presents to the applied field. Based on the full sample size, the magnetic current density J_c is calculated using the relationship for a plate in a perpendicular field: $J_c = 20\Delta M / (a - a^2/3b)$. J_c versus magnetic field up to 9 T for the samples at 5 K, 10 K, 20 K, and 30 K has been measured. Due to flux jumping, the low field J_c at values below 10 K cannot be measured. The results are displayed in table 1.

Example 2: Synthesis of MgB_{2-x}(SiC)_x using nanoparticle SiC to react with Mg and B

MgB_{2-x}(SiC)_x samples were prepared by a reaction in-situ method. Powders of magnesium (99% purity) and amorphous boron (99% purity) were thoroughly mixed with SiC additive, to prepare various samples with the following ratios of MgB_{2-x}(SiC)_x: X = 0, 0.02, 0.05, 0.1, 0.15, 0.2, 0.5, 1.0, 1.5 and 2.0. The mixed powders were loaded into Fe tubes. The composite tubes were groove-rolled, sealed in a Fe tube and then directly heated at preset temperatures to 900°C, for 3 hours in the presence of flowing high purity Ar. This was followed by quenching to liquid nitrogen

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temperature.

Example 3: Synthesis of $MgB_{2-x-y}Si_xC_y$ using Mg, B, Si and C powders

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MgB_2 pellet samples were prepared by a reaction in-situ method. Powders of magnesium (99% purity) and amorphous boron (99% purity) were well mixed with SiC additive with the ratio of $Mg+B_{2-x}(SiC)_x$ where $x = 0, 0.02, 0.05, 0.1, 0.15, 0.2, 0.5, 1.0, 1.5$ and 2.0 . The mixed powders were loaded into Fe tubes. The composite tubes were groove-rolled, sealed in a Fe tube and then directly heated at preset temperatures to $900^\circ C$, for 3 hours in flowing high purity Ar. This was followed by quenching to liquid nitrogen temperature.

Example 4: Synthesis of $MgB_{2-x-y}Ti_xC_y$ using Mg, B, TiC powders

20 MgB_2 pellet samples were prepared by a reaction in-situ method. Powders of magnesium (99% purity) and amorphous boron (99% purity) were well mixed with TiC additive with the ratio of $Mg+B_{2-x}(TiC)_x$ where $x = 0, 0.05, 0.1, 0.15, 0.2, 0.5, 1.0$. The mixed powders were loaded into Fe tubes. The composite tubes were groove-rolled, sealed in a Fe tube and then directly heated at preset temperatures to $950^\circ C$, for 3 hours in the presence of flowing high purity Ar. This was followed by furnace cooling to room temperature.

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Example 5: Synthesis of $MgB_{2-x}C_x$

Polycrystalline samples of $MgB_{2-x}C_x$ were prepared through a reaction in-situ method. High purity powders of magnesium (99% purity), amorphous boron (99% purity) and carbon nano-particles (with a particle size of about 20nm) were weighed out according to the nominal atomic ratio of MgB_2 .

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xC_x with $x = 0, 0.05, 0.1, 0.2, 0.3, 0.4$ and well-mixed through grinding. The powders were pressed into pellets of 10 mm in diameter and 3 mm in thickness using a hydraulic press. The pellets were sealed in Fe tubes, then heat 5 treated at 770 °C for 30min in flowing high purity Ar. This was followed by a furnace cooling to room temperature. An un-doped sample was also made under the same conditions for use as a reference sample. The phase and crystal structure of all the samples was obtained from X-ray 10 diffraction (XRD) patterns using a Philips (PW1730) diffractometer with $\text{CuK}\alpha$ radiation. Si powder was used as a standard reference to calculate the lattice parameters. The results show that both the a -axis lattice parameter and the T_c decreased monotonically with increasing doping 15 level. For the sample doped with the highest nominal composition of $x=0.4$ the T_c dropped only 2.7K. The nano-C-doped samples showed an improved field dependence of the J_c compared with the undoped sample over a wide temperature range. The enhancement by C-doping is similar to that of 20 Si-doping but not as strong as for nano-SiC doped MgB₂. X-ray diffraction results indicate that C reacted with Mg to form nano-size Mg_2C_3 and MgB_2C_2 particles. Nano-particle inclusions and substitution, both observed by transmission electron microscopy, are proposed to be responsible for 25 the enhancement of flux pinning in high fields.

Example 6: Synthesis of $\text{MgB}_{2-x}\text{Si}_x$

Polycrystalline samples of $\text{MgB}_{2-x}\text{Si}_x$ were prepared through a 30 reaction in-situ method. High purity powders of magnesium (99% purity), amorphous boron (99% purity) and silicon nano-particles (with a particle size of about 20-40nm) were weighed out according to the nominal atomic ratio of 35 $\text{MgB}_{2-x}\text{Si}_x$ with $x = 0, 0.05, 0.1, 0.2, 0.3, 0.4$ and well-mixed through grinding. The powders were pressed into pellets of 10 mm in diameter and 3 mm in thickness using a hydraulic press. The pellets were sealed in Fe tubes, then

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heat treated at 800-900°C for 30min in flowing high purity Ar. This was followed by a furnace cooling to room temperature. An un-doped sample was also made under the same conditions for use as a reference sample.

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Although the above embodiments specifically relates to magnesium diboride superconducting material it is possible that silicon carbide codopant could be useful for other superconducting materials.

10 A suitable process for utilising the superconducting material of the present invention would be as follows:

- Mixing powders of precursor materials as discussed above.
- Ball milling the mixture to achieve homogeneity.
- 15 • Loading the mixture in to metal or alloy dies, containers, tubes for forming and shaping, the metals including iron, stainless steel, nickel and various alloys.
- Making the mixtures or the composites in to bulk, wires, tapes and various shaped articles and devices through mechanical deformation including pressing, drawing, rolling, swaging and casting etc.
- 20 • Sintering the composites at temperatures of 650°C to 1000°C for 10 minutes to 10 hours, preferably at 800 to 950°C for 10 minutes to 3hours.
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Modifications and variations as would be apparent to a skilled addressee are deemed to be within the scope of the present invention.

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